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Partitioning of Toxic Organic Compounds on Municipal Wastewater Treatment Plant Solids

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16. ABSTRACT

Fundamental aspects of partitioning of toxic organic compounds on municipal wastewater treatment plant solids have been investigated. Sorption on wastewater solids was not affected by solids-to-liquid ratio. Kinetic data on sorption showed an initial rapid uptake followed by a slower rate over an extended period of time. Freeze-dried solids did not exhibit the same sorption characteristics as viable biomass.

A correlation between sorption of toxic organics on wastewater treatment plant solids and the octonal/water partition coefficient has been established. The correlation should be useful for estimating the removal of toxic compounds in wastewater treatment plants by the sorption mechanism.

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PARTITIONING OF TOXIC ORGANIC COMPOUNDS ON MUNICIPAL WASTEWATER TREATMENT PLANT SOLIDS

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INTRODUCTION

Partitioning on solids is one of the fundamental processes controlling the removal of toxic organic compounds in municipal wastewater treatment plants. The literature provides ample evidence that organics may accumulate in sludges at concentrations several orders of magnitude greater than the influent concentrations. Compounds which have a special affinity for sorption include pesticides, phthalates, and polynuclear aromatic hydrocarbons which have shown concentration factors of 13-13,000 (1,2,3). The fate of hydrophobic organic pollutants in water systems is highly dependent upon their sorptive behavior. The degree of sorption not only affects a chemical's mobility, but also is a dominant factor in fate processes such as volatilization and biodegradation.

Since interphase partitioning phenomena are one of the major steps in the removal of organics from the bulk aqueous flow in wastewater treatment plants, it is important that

both quantitative and qualitative information about the sorption process be obtained. These results will enable the prediction of removal by wastewater solids, provide a basis for modeling the process, and provide a data base to correlate sorption on wastewater solids with other sorption processes and with molecular properties.

In dilute systems typical of most environmental situations, the partition coefficient Kp for sorption on wastewater solids can be defined as follows:

$$K_p = C_s/C_L \qquad (1)$$

where: C_s = concentration of pollutant in the solid phase and C_L = concentration of pollutant in the liquid phase.

The usual method for measuring partition coefficients is to determine a sorption isotherm. Slurries of the wastewater solids are prepared in different initial concentrations of the toxic organic chemical being studied. After the systems reach equilibrium, the concentration in both the solution and solid phases are measured (solid phase concentration can be calculated based on the difference between the initial concentration added and the solution concentration at equilibrium). Data are fitted to the Freundlich equation which can be written:

$$X/M = KC_e^n \qquad (2)$$

where: $X = C_o - C_e$ which is the amount of solute sorbed from a given volume of solution
C_o = initial concentration of solute added
C_e = concentration of solute at equilibrium
M = weight of solids added to the solution
K and n = empirical constants

Data are fitted to the logarithmic form of Equation (2) which can be written as follows:

$$\log X/M = \log K + n \log C_e \qquad (3)$$

For dilute solutions this equation yields a straight line with a slope of n and an intercept equal to the value of K (X/M at $C_e = 1.0$) when X/M is plotted as a function of C_e on logarithmic paper. The intercept is an indicator of sorption capacity and the slope of sorption intensity. K is often referred to as the sorption or adsorption coefficient. To obtain the partition coefficient K_p) when X/M is in mg/gm and C_e is in mg/l,

the following equation is used:

$$Kp = \frac{X/M}{C_e/1000} \qquad (4)$$

(Equation (4) also applies to the case where X/M is in µg/gm and C_e is in µg/l)

The Freundlich equation is a useful method for the treatment of sorption data. Since $X = C_o - C_e$, substitution of $C_o - C_e$ for X in Equation (2) yields:

$$(C_o - C_e)/M = KC_e^n \qquad (5)$$

which gives removal of toxic organic compound as a function of solids concentration.

MATERIALS AND METHODS

Reagents

Organic compounds were obtained from Eastman Kodak Co., Rochester, NY; Aldrich Chemical Company, Inc., Milwaukee, WI; and Chem Service Inc., West Chester, PA.* All chemicals were the purest available and were used as received from the supplier.

Analytical Methods

Analytical methods for analysis of the selected organic compounds used in the present study are summarized in Table I.

Volatile chlorinated organics were analyzed using a Tracor LSC-2 sample concentrator and a Tracor 222 gas chromatograph equipped with a Tracor 700 Hall electrolytic conductivity detector. Pesticides and the cresol were analyzed using a Varian Model 3700 gas chromatograph equipped with a Varian CDS-111 chromatographic data system. Dyes were determined with Beckman Model 25 spectrophotometer equipped with a clinical sipper system.

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Table I. Summary of Analytical Methods Used for Analysis of Toxic Organic Compounds

Compounds	Analytical Methods	Conditions
Methylene chloride Chloroform 1,1-dichloroethylene Carbon tetrachloride Tetrachloroethylene Chlorobenzene	purge and trap gas chromatography	Texas trap; 3% Carbowax 1500 on chromosorb column; Hall detector
Lindane Heptachlor epoxide Dieldrin	hexane extraction gas chromatography	1.5% SP-2250/ 1.9% SP-2401 on Supelcoport column election capture detector
2,4-dinitro-o-cresol	freon extraction gas chromatography	1% SP-1240 DA on Supelcoport column flame ionization detector
Methylene blue dye Azo dye 88 Azo dye 151	visible spectroscopy	665 nm 505 nm 512 nm

Wastewater Solids Preparation

Wastewater solids were collected from municipal wastewater treatment plants within a fifty mile radius of the Water Engineering Research Laboratory, Cincinnati, Ohio. Wastewater plants were selected to include a wide range of domestic/industrial contribution to the total plant flow. Samples of primary sludge, mixed liquor solids, and digested sludge were collected in a series of five gallon carboys. Solids were removed by centrifugation in a perforate bowl centrifuge. Analysis of the sludge or mixed liquor and the centrate showed solids capture of approximately 95±3% by centrifugation. The collected solids (approximately 10% on a dry weight basis) were stored

at 4°C until used. No visible change was observed during the cold storage period of 30-90 days required for completion of experimental testing for a given wastewater solid system.

EXPERIMENTAL PROCEDURE

Experimental Protocol Development

Preliminary Studies

A standard experimental protocol for determination of the partition coefficient or sorption capacity of wastewater solids for toxic organic compounds has not been developed. Reports in the literature by different investigators are in direct conflict with regard to several fundamental concepts related to the partitioning process. In order to resolve these issues and establish a basis for partition measurements, several preliminary studies were conducted. Key issues to be resolved include: (1) effect of solids-to-water ratio on the partition measurement; (2) rate of attainment of equilibrium; and (3) use of viable versus non-viable solids in the isotherm procedure.

Several reports in the literature state that the equilibrium partition coefficient (K_p) decreases as the ratio of solids-to-water increases (4,5,6). These observations challenge the classical view of the sorption process. The two procedures available for sorption capacity measurements involve the use of a constant concentration of compound with varied amounts of solids or the use of a varied concentration of compound with a constant amount of solids. If solids-to-water ratio affect the measurement of partition coefficients, the first method described should not be used. In order to resolve this issue, a preliminary set of isotherms were measured using both methods. The test involved determination of the sorption or partition coefficient for methylene blue dye using mixed-liquor solids.

In the first of the two procedures, a constant methylene blue dye concentration of 10.0 mg/l was used with mixed-liquor solids concentrations of 0, 0.05, 0.10, 0.25, 0.30, and 0.5 g/l on a dry weight basis. These solid doses selected gave a 10-fold variation in solids-to-liquid ratio and are typical of the conditions used to measure partition coefficients. In the second procedure, a constant mixed-liquor solids concentration of 0.1 g/l dry weight was used in conjunction with methylene blue dye concentrations of 0, 2.5, 5.0, 7.5, 10.0,

and 20.0 mg/l. Methylene blue dye was chosen for the preliminary test because it is non-biodegradable, non-volatile, and easily determined by visible spectroscopy at 665 nm. Test samples containing the specified solids and dye concentration were prepared and equilibrated for 8 hours. Solids were separated by centrifugation and the concentration of dye measured. The isotherms obtained by both methods are shown in Figure 1 where X/M is plotted as a function of C_e according to the logarithmic form of the Freundlich equation.

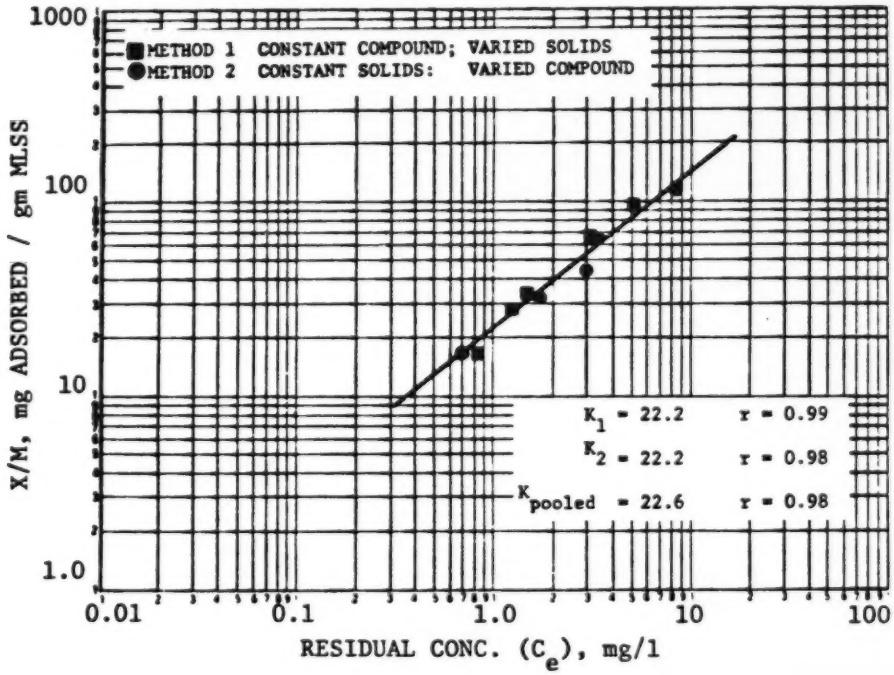


Figure 1. Comparison of two isotherm methods for sorption capacity measurements.

The sorption coefficients (K values) calculated from the least squares treatment of the separate isotherm data were the same for both methods (22.2 mg/gm). The pooled data yielded a K value of 22.6 mg/gm in excellent agreement with the two individual isotherm values. These data indicate that the sorption coefficient and the partition coefficient remain constant over the range of solid-to-water ratios used in this study. It has been emphasized that if precautions are taken to eliminate or

account for non-settling (or non-filtrable) microparticles which remain in the aqueous phase during sorption tests, the partition coefficient does remain constant (7) in agreement with the present study. Even though either method can be used to experimentally determine sorption and partition coefficients, the second method discussed was selected for use in the present study. The choice was based on the desire to maintain a constant solids level in the samples during the solids' separation step by centrifugation or filtration prior to analysis. A second reason for the use of constant solids in the sorption test is that frequently measurable levels of a given test compound will be found in the wastewater solids as a natural background. If this occurs, it is easier to correct analytical values of the equilibrium concentrations in the samples by a constant amount.

A great deal of confusion exists in the literature regarding the rate of attainment of sorption equilibrium. Sorption kinetics for phenol and 1,4-dichlorobenzene were reported to be exceedingly fast (equilibrium in <4 minutes) (8). In the case of polynuclear aromatic hydrocarbons on natural sediments 35 to 60% of the equilibrium sorption capacity was achieved in minutes (mixing limited) with apparent equilibrium after a few hours (9).

Based on the conflicting reports in the literature on the rate of attainment of equilibrium, kinetic measurements on the sorption of methylene blue dye on mixed-liquor solids were made to determine the proper contact time for experimental purposes. Mixed-liquor solids from three diferent municipal wastewater treatment plants were collected. Rate of sorption of methylene blue dye from a 10 mg/L solution was measured at 0.1,0.2 and 0.5 g/L dry weight solids. All three mixed-liquor samples produced similar results. Data for the mixed-liquor solids from the same wastewater treatment plant used in the isotherm runs are presented in Figure 2.

The kinetic data for the sorption of methylene blue dye are in agreement with the two box model proposed for hydrophobic pollutants in water (10). The two box model described sorption dynamics in terms of a rapid or "labile" exchange and a highly retarded or "non-labile" sorption requiring days to weeks to occur. In the case of methylene blue dye and 0.5 g/l mixed-liquor solids dose, 92% of the compound was sorbed in approximately 30 minutes, while the remainder required several hours. In order to minimize biodegradation during the sorption test, the remaining organic compounds were equilibrated for 6 hours. This avoided overnight contact and permitted samples to be separated from the solids the same day.

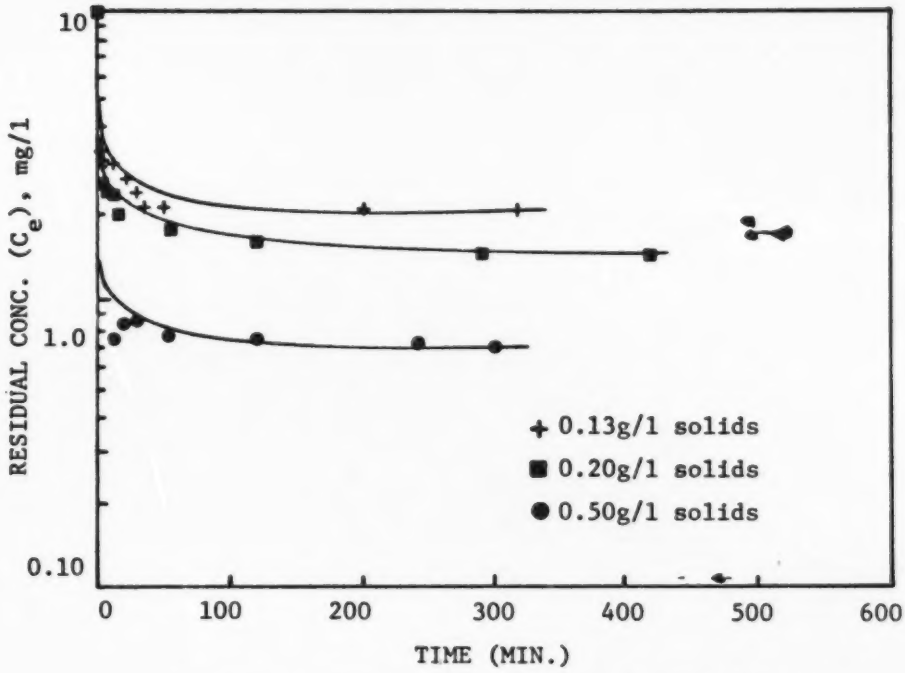


Figure 2. Kinetics of sorption of methylene blue dye on mixed-liquor solids.

The third issue to be resolved in preliminary studies involved methods to eliminate biological degradation of test compounds during the partitioning process. It is obvious that other removal mechanisms such as biodegradation and volatilization would have to be controlled if accurate sorption data were to be obtained. Otherwise, measurements of changing aqueous phase compound concentrations would include the effects of all removal mechanisms resulting in apparent sorption capacities greater than actually achieved. Volatilization was eliminated from sorption measurements by using completely filled containers with zero head space. The impact of biodegradation on the sorption measurements was more difficult to control. Lyophilization followed by dry heat treatment has been reported to produce biological solids which had flocculation and settling properties which were indistinguishable from live biomass (8). These authors claimed the batch experimental technique with nonviable biomass appeared to give realistic results compared to tests with viable biomass. Based on the reported successful

use of lyophilization for control of biological activity, this technique was evaluated. A batch of mixed-liquor solids was collected and separated from the supernatant in a perforate bowl centrifuge. The moist solids were divided into two portions. One portion was freeze-dried and the other was stored at 4°C. Sorption isotherms were measured using 2,4-dinitro-o-cresol. The isotherms were conducted at pH 3.0 for two reasons. First, the low pH would inhibit or eliminate biological activity in the test with viable biomass. Secondly, the decrease in pH would convert the 2,4-dinitro-o-cresol to the undissociated (sorbable) form (pKa = 4.35). Results of the isotherm comparison are shown in Figure 3.

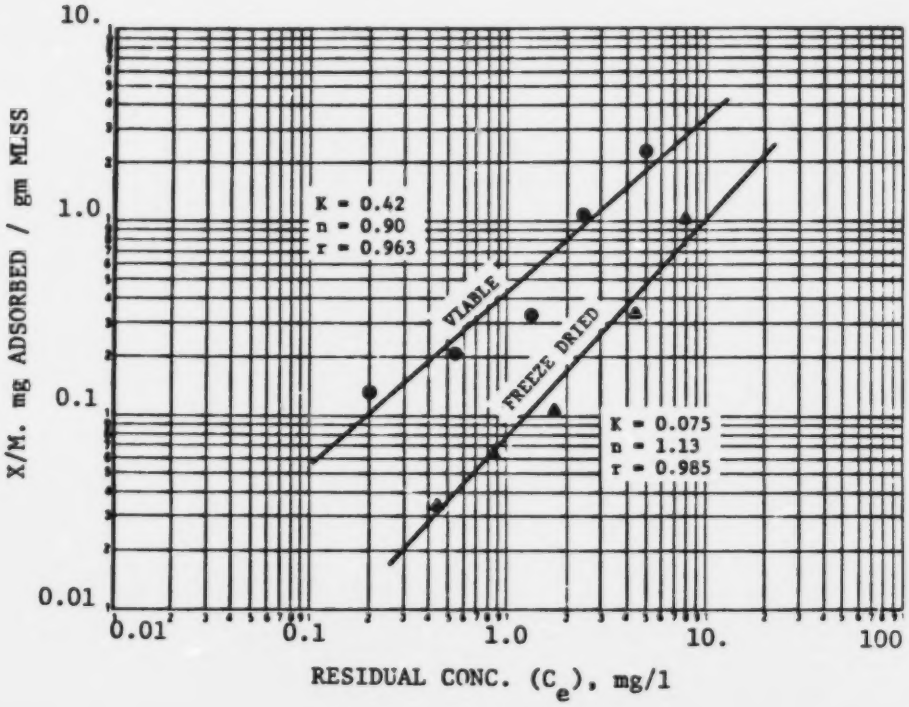


Figure 3. Comparison of freeze-dried and viable biomass for sorption of 2,4-dinitro-o-cresol at pH 3.0.

The use of freeze-dried solids in the isotherm procedure was not acceptable based on the results shown in Figure 3. The viable biomass clearly has a significantly greater sorption capacity than the freeze-dried material. A similar result has been reported for sorption of azo and triphenylmethane dyes on microbial populations (11).

Additional studies are under way to develop methods to control biological activity during the partitioning process. Preliminary data suggest that pH adjustment to 3.0 may effectively eliminate biological activity without significantly affecting the partitioning process for neutral and acidic compounds.

Sorption or Partitioning of Selected Toxic Organics

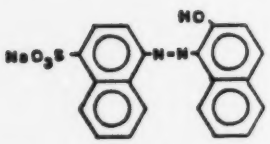
Based on the results of the preliminary tests, additional sorption isotherms were measured using viable mixed-liquor solids with a 6-hour contact time. Compounds selected for experimental use were either resistant to biodegradation or were shown not to significantly degrade during the course of the isotherm test by specific analysis on whole samples (both aqueous and solid phases). Chemical structures for the toxic organic compounds investigated to date are shown in Figure 4.

DISCUSSION OF RESULTS

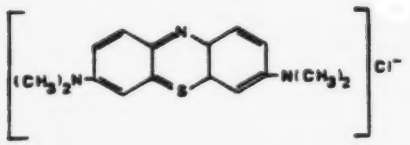
The isotherms obtained for the toxic organic compounds studied showed a wide range of sorption capacities. Relevant Freundlich parameters for sorption on mixed-liquor solids are summarized in Table II.

Sorption coefficients (K values) ranged from 0.062 mg/gm for methylene chloride to an extrapolated value of 38.9 mg/gm for dieldrin at equilibrium concentrations of 1.0 mg/l. Slopes (n values) were generally close to 1.0 and ranged from 0.31 to 1.15 for sorption on mixed-liquor solids. Slopes for sorption on soils have been reported to range from a low of 0.3 to a high of 1.7 (12). Compiled data on 26 chemicals (mostly pesticides) had a mean value of n of 0.87 with a coefficient of variation of ±15% (13). (If a measured value is not available, it is frequently assumed to be equal to 1.0.) Excellent correlation coefficients (r values) were obtained for all the isotherms measured.

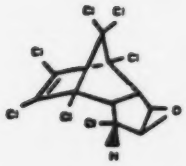
In soil and sediment sorption, the extent to which an organic chemical partitions itself between the solid and solution phases is expressed in terms of a parameter K_{OC}, which is largely independent of the properties of the soil or sediment.



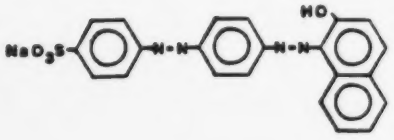
Azo Dye 88



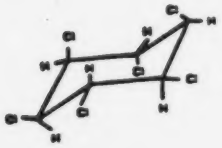
Methylene Blue



Heptachlor Epoxide

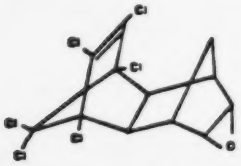


Azo Dye 151

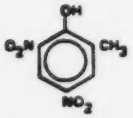


Lindane

Methylene Chloride



Dieldrin

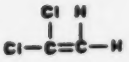


2,4-dinitro-o-cresol

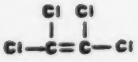
Chlorobenzene

Carbon tetrachloride

Chlorofrom



1,1-dichloroethylene



Tetrachloroethylene

Figure 4. Structures of organic compounds used in sorption studies.

Table II. Summary of Freundlich Parameters For Sorption of Toxic Organics on Mixed-Liquor Solids

Compound	K,(mg/gm)[a]	n[b]	r[c]
Methylene chloride	0.062	1.15	0.92
Chloroform	0.094	0.90	0.98
1,1-dichloroethylene	0.150	0.71	0.96
Carbon tetrachloride	0.597	0.31	0.97
Chlorobenzene	0.330	0.47	0.91
2,4-dinitro-o-cresol	0.420	0.90	0.96
Tetrachloroethylene	0.970	1.16	0.98
Lindane[d]	0.762	1.00	0.99
Heptachlor epoxide[d]	17.9	1.09	0.98
Dieldrin[d]	38.9	1.11	0.99
Methylene blue dye	22.6	0.82	0.98
Azo dye 151	8.30	1.00	0.99
Azo dye 88	1.35	0.65	0.99

[a] K = sorption coefficient = X/M at C_e = 1.0 mg/l
[b] n = slope of isotherm plot
[c] r = correlation coefficient
[d] K values extrapolated to C_e = 1.0 mg/l for comparison purposes.

Once K has been determined from the Freundlich equation, it is divided by % organic carbon (oc) contained in the soil or sediment and multiplied by 100 to obtain K_{oc}. In the case of wastewater solids used in the present study, organic carbon was based on weight loss on ignition at 600°C and is defined by the following equation:

$$K_{om} = K \cdot \frac{100}{\% \text{ VSS}} \qquad (6)$$

where K_{om} = amount of solute or pollutant sorbed per unit weight of organic matter in solids.
VSS = volatile suspended solids.

Sorption data are usually correlated based on organic carbon or organic matter. Therefore, the sorption coefficient, K, must be converted to K_{om} for correlation purposes. Thus, we can

define a corrected partition coefficient by the following equation:

$$K_p' = \frac{K_{om}}{C_e/1000} \quad (7)$$

Emphasis on the environmental impact of toxic organic chemicals has resulted in increased reliance on the physical-chemical properties of these compounds to assess their environmental behavior. One such parameter, the octanol/water partition coefficient, (K_{ow}) has proved useful as a means for predicting soil adsorption (14), biological uptake (15), lipophilic storage (16), and biomagnification (17, 18, 19, 20). K_{ow} is defined by the following equation:

$$K_{ow} = C_o/C_w \quad (8)$$

where C_o = equilibrium concentration of organic compound in octanol layer

C_w = equilibrium concentration of organic compound in water layer

Based on the soil-sediment literature, an attempt was made to correlate sorption on wastewater solids with the octanol/water partition coefficient for the compounds used in the present study. Sorption coefficients in Table II were converted to K_{om} values using Equation 6. The K_{om} values obtained were used to calculate corrected partition coefficients (K_p' values) using Equation 7. The values obtained are summarized in Table III along with the corresponding octanol/water partition coefficients.

A simple linear regression of log K_p' on log K_{ow} yielded a correlation coefficient (r) of 0.985. A plot of log K_p' versus K_{ow} is shown in Figure 5. The following equation was obtained for the relationship between partitioning on wastewater solids and octanol/water partition coefficient:

$$\log K_p' = 1.06 + 0.60 \log K_{ow} \quad (8)$$

If K_{ow} is given in numerical form, the equation can be written as follows:

$$K_p' = 11.6(K_{ow})^{0.6} \quad (9)$$

Table III. Summary of Values Used to Correlate Sorption on Mixed-Liquor Solids with Octanol/Water Partition Coefficient

Compound	K_{om} (a)	K_p'	log K_p'	log K_{ow} (b)
Methylene chloride	0.067	67	1.83	1.26
Chloroform	0.117	117	2.07	1.97
1,1-dichloroethylene	0.185	185	2.27	2.13
Carbon tetrachloride	0.735	735	2.87	2.64
Chlorobenzene	0.407	407	2.61	2.84
2,4-dinitro-o-cresol	0.590	590	2.77	2.85
Tetrachloroethylene	1.11	1110	3.04	2.88
Lindane	2.57	2570	3.41	3.85
Heptachlor epoxide	15.33	15,330	4.19	5.40
Dieldrin	25.16	25,160	4.40	5.48

(a) K_{om}: units are mg/gm volatile suspended solids at Ce = 1.0 mg/l for first seven compounds listed and µg/gm volatile suspended solids at C_e = 1.0 µg/l for the three pesticides.

(b) K_{ow}: values were taken from reference (21) or were calculated by fragment method (22).

Limited tests have been completed on the sorption of toxic organics on primary sludge and digested sludge. Data for two compounds are presented in Table IV.

Table IV. Comparison of Partition Coefficients (K_p (a) Values) for Sorption of Toxic Organics on Wastewater Solids

Compound	Primary Sludge	Mixed-Liquor	Digested Sludge
1,1-dichloroethylene	0.20	0.15	0.17
Chlorobenzene	0.35	0.33	0.38

(a) K_p: units are mg/gm solids

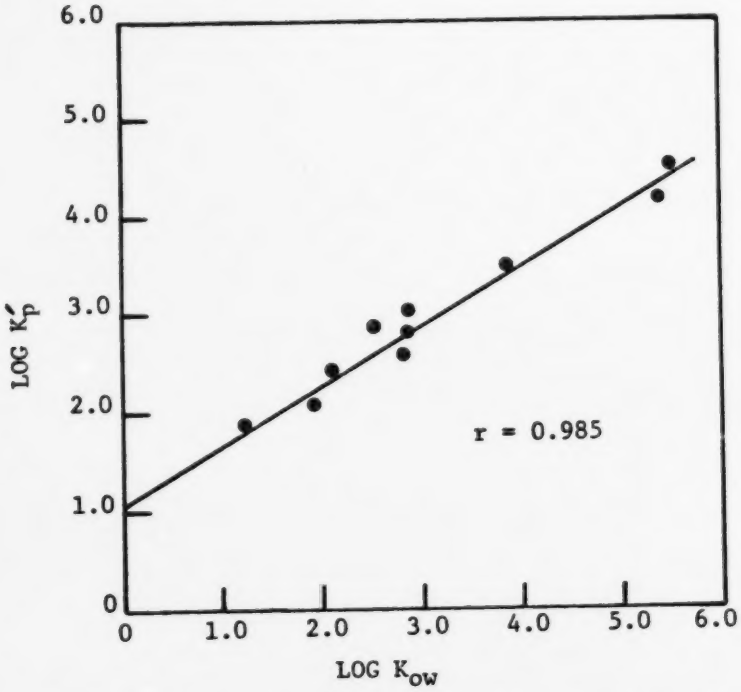


Figure 5. Correlation of partition coefficients for sorption on mixed-liquor solids with octanol-water partition coefficients.

Based on limited data, it appears that the relationship between K_p' and K_{ow} established for mixed-liquor solids is also valid for other municipal wastewater treatment plant solids. Additional isotherms are being measured to expand the data base and validate the relationship for other solids.

SUMMARY AND CONCLUSIONS

Preliminary studies have shown that partitioning on municipal wastewater treatment plant solids was not affected by solids-to-liquid ratio. Kinetic data on sorption of toxic organic compounds on wastewater treatment plant solids showed an initial rapid uptake followed by a slower rate over an extended period of time. Freeze-dried solids did not exhibit the same sorption characteristics as viable biomass.

A correlation between sorption of toxic organics on wastewater treatment plant solids and octanol/water partition coefficient has been established. The relationship should be useful for estimating the removal of toxic organic compounds in municipal and industrial wastewater treatment plants by the sorption mechanism. The correlation also provides a basis for predicting concentrations of toxic compounds in various sludges provided the equilibrium concentration in the aqueous phase is known (or assumed).

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